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**EP 0 249 770 B1**

## Description

Suspension concentrate or aqueous flowable pesticidal compositions are concentrated suspensions of water insoluble pesticides and mixtures of pesticides in aqueous systems. The present invention relates to stable pendimethalin compositions.

Pendimethalin is a dinitroaniline that exists in polymorphic form, both in orange crystal form and yellow crystal form. U.S. Patents 4,082,537 and 4,150,969 address pendimethalin's unique formulation problems and provide ways to avoid formation and/or presence of the larger, orange crystal form. It is believed that the presence of pendimethalin in the orange macrocrystal form results in large elongated crystals in final formulations.

In order to formulate stable wettable powder compositions of pendimethalin, these two patents disclose utilizing a stabilized pendimethalin, stabilized by the addition of either sodium dialkyl ( $C_6-C_8$ ) sulfosuccinate or an ethoxylated  $\beta$ -diamine. This technique maintains pendimethalin in the yellow crystal form which does not favor the formation of large elongated crystals in formulated product for the preparation of wettable powders. However, these patents fail to disclose ways to formulate pendimethalin as a stable suspension concentrate composition or aqueous flowable composition with orange crystal forms, which form is favored.

These aqueous compositions frequently contain about 10% to 80%, by weight of a solid pesticide or mixture of solid pesticides, thereby providing a method for handling those pesticides which are relatively water insoluble in an aqueous medium. Since these types of compositions have the desirable characteristics of a thick liquid, they may be poured or pumped. Thus, some of the problems, like dusting that is possible in solid compositions of wettable powders and granulars, are avoided. Further, these aqueous-based concentrates also have the added advantage of not requiring the use of organic solvents, often present in emulsifiable concentrates.

For these reasons, it is desirable to formulate pesticides into suspension concentrates or aqueous flowables. However, such formulations have their own problems such as gelling, caking and settling, as well as problems because of the physical and chemical characteristics of the pesticide or mixture of pesticides. Pendimethalin is one of these pesticides that is somewhat difficult to formulate.

Oftentimes, when pendimethalin in the orange macrocrystal form is found in compositions, very large, elongated crystals (about 3000  $\mu m$  in length) appear in final product, resulting in instability, difficulty in processing and unreliability of usage. Thus, formulating compositions wherein these elongated crystals do not develop is crucial to stability and necessary to obtain even distribution of active compound for application.

The problems associated with the development of suspension concentrate compositions containing low melting active ingredients, alone or in combination with higher melting active ingredients, are described in German Patent Application DE 3302648 A1. German Patent Application DE 3302648 A1 tries to deal with the problems of an aqueous mixed dispersion of a low melting active ingredient in a solvent of phthalic acid  $C_1-C_{12}$  alkyl esters in combination with an aqueous suspension concentrate containing one or more active ingredients as an alternative to a suspension concentrate containing low melting active ingredients, such as pendimethalin[N-(1-ethyl-propyl)-2,6-dinitro-3,4-xylidine]. The reason for the alternative approach of that application is the inability to prepare stable suspension concentrates by various techniques, including those of European Patent Application 0 33291.2 That EPO application describes insecticidal suspension concentrate compositions of phosalone and adjuvants which may be prepared with molten insecticide. These references do not address ways helpful to the development of pendimethalin compositions which utilize the orange crystal form to produce a stable and evenly efficacious product.

It is an object of the present invention to provide stable aqueous concentrate compositions of the low melting pesticide, pendimethalin, in the orange crystal form in order to avoid the problem of formation of large elongated crystals of formulated pendimethalin.

It is a further object of this invention to provide such stable aqueous suspension concentrate compositions of pendimethalin having a ratio of orange crystal to yellow crystal of 4:96 to totally (100%) orange crystal form.

Furthermore, an additional object of the present invention is to provide methods for preparing such stable aqueous suspension concentrate compositions.

These and other objects will become more apparent by the detailed description of the invention provided herein.

The present invention provides an aqueous suspension concentrate composition consisting essentially of 5.0 % to 50.0 % pendimethalin having at least 4 % and up to 100 % orange crystal form; 3.0 to 30.0 % coformulants and 20.0 % to 92.0 % water, obtainable by admixing, on a weight to volume basis, 5.0 % to 50.0 % pendimethalin having 4 % orange crystal form to 96 % yellow crystal form to 100 % orange crystal

form; in an aqueous solution containing some of the coformulants such as a surfactant, a dispersing agent or wetting agent and an antifoaming agent; milling the resulting mixture, wherein the average particle size of suspended particles is brought to less than 20  $\mu\text{m}$ ; admixing the remaining coformulants, such as a thickening agent, a suspending agent, an antifreezing agent, a preservative and an additional surfactant; and packaging the resulting aqueous suspension concentrate composition.

#### DETAILED DESCRIPTION OF THE INVENTION

Preferred stable aqueous suspension concentrate compositions of pendimethalin comprise on a weight to volume basis, 5.0% to 50.0% pendimethalin, wherein 4.0% of the pendimethalin crystals are the orange crystal form, most preferred 10.0% or more of the pendimethalin crystals are the orange crystal form. Further, the compositions of the present invention also comprise, on a weight to volume basis, 0.5% to 1.0% antifoaming agents; 2.0% to 20.0% antifreezing agent; 2.0% to 20.0% surfactants dispersing agents, wetting agents, or mixtures thereof; 0.05% to 3.0% thickening agents; 0.01% to 1.0% preservatives; and the remainder water to total the composition to 100%.

#### Coformulants

Surfactants (including dispersing agents and/or wetting agents) suitable in the aqueous suspension compositions of the invention containing solid pendimethalin include: ethylene oxide/propylene oxide condensates; alkyl, aryl- and aryl, aryloxyates and derivatives thereof; lignosulfonates; cresol- and naphthalene-formaldehyde condensates and sulfonates; polycarboxylates and derivatives thereof; and mixtures thereof.

In general, anionic polymerics, such as cresol formaldehyde condensates and their sulfonates, naphthalene formaldehyde condensates and their sulfonates and lignosulfonates have been found to minimize crystal formation during storage and as such are most preferred.

Suspending agents such as polysaccharide gums like Xanthan gum, guar gum; gum arabic and cellulose derivatives and the like are suitable for addition in amounts of 0.02% to 3.0%, on a weight to volume basis.

Preservatives to prevent microbial spoiling in the compositions of the invention are included as necessary. One example is a 38% formaldehyde solution. Other preservatives include methyl or propyl parahydroxybenzoate, 2-bromo-2-nitro-propane-1,3-diol, sodium benzoate, glutaraldehyde, O-phenylphenol, benzisothiazolinones, 5-chloro-2-methyl-4-isothiazolin-3-one, pentachlorophenol, 2-4-dichlorobenzylalcohol, or mixtures thereof and others known to those in the art. Silicic antifoaming agents are useful in the present compositions.

Antifreezing agents such as ethylene glycol, propylene glycol, other glycols, glycerine or urea may be added to the aqueous suspension concentrate compositions. Additional surfactants, preservatives and thickening agents, such as clays, precipitated silicas, polyvinyl alcohol, polyvinyl-pyrrolidone, polyacrylamides and the like, may then be added, as can higher melting active components or a suspension concentrate containing other active components.

#### Process of Manufacturing

Surprisingly, it has been found that stable aqueous suspension concentrate compositions of pendimethalin are prepared with at least 4.0% of the pendimethalin in the orange crystal form, contrary to what has been reported previously. In the present invention pendimethalin even in the large orange crystal form may be used to formulate stable aqueous suspensions. Further, up to 100% of the pendimethalin may be present as the orange crystal form when preparing the compositions of the invention, whereas only the yellow crystal form was favored to avoid large elongated crystals of formulates of pendimethalin.

Thus, aqueous suspension concentrate compositions of pendimethalin alone or in combination with a second higher melting pesticide(s) comprise on a weight to volume basis, 5.0% to 50.0% of pendimethalin containing 4.0% to 100% of the orange crystal form; 3.0% to 30.0% coformulants as described hereinabove; and the remainder water to total the composition to 100%.

The compositions of the present invention are readily prepared by adding solid pendimethalin to water that may contain some of the coformulants, such as the antifoaming agents and/or suspending agents; milling at ambient temperatures the resulting solid-aqueous mixture to obtain an average particle size of less than 20  $\mu\text{m}$ , preferably 2  $\mu\text{m}$  to 10  $\mu\text{m}$  and then adding, if desired, other coformulants such as thickening agents, antifreezing agents, surfactants, dispersing agents, wetting agents, suspending agents and

preservatives.

The following examples further illustrate the present invention and are not limitative thereof.

# EXAMPLES 1-9

## Preparation of stable aqueous suspension concentrate compositions of pendimethalin

An aqueous solution containing surfactant(s) and antifoaming agents, and if desired, water soluble pesticides is prepared at ambient temperatures. Solid pendimethalin, having a minimum of 4% of the orange crystal form, is added, and any other solid second pesticide having a melting point greater than 70°C may also be added.

This is milled to achieve the desired average particle size of suspended particles of less than 20 µm, preferably less than 6 µm (2 µm to 6 µm). Then thickening agents, suspending agents, antifreezing agents, preservatives and additional surfactants are admixed with the resulting mixture. Finally, this resulting stable aqueous suspension concentrate composition is packaged.

Utilizing the above procedure yields the stable aqueous suspension concentrate compositions listed in Table I.

TABLE I  
Stable aqueous suspension concentrate compositions (% w/v)

Composition	Example								
	1	2	3	4	5	6	7	8	9
Pendimethalin - (orange/yellow ratio)	23.6 (32/68)	23.6 (32/68)	23.6 (32/68)	23.6 (24/76)	23.6 (16/84)	23.6 (8/92)	23.6 (4/96)	20.0 (46/54)	44.8 (100%)
Isoproturon	23.6	23.6	23.6	23.6	23.6	23.6	23.6	-	-
Chlortoluron	-	-	-	-	-	-	-	30.0	-
Na <sup>+</sup> cresol-formaldehyde sulphonated condensate	3.0	3.0	-	3.0	3.0	3.0	3.0	3.0	-
Na <sup>+</sup> naphthalene sulfonated condensate	-	-	-	-	-	-	-	-	3.0
Ethylene oxide - propylene oxide copolymer	2.0	-	-	-	-	-	-	2.0	-
Na <sup>+</sup> lauryl sulphate	-	0.5	-	0.5	0.5	0.5	0.5	-	-
Triethanolamine salt of polyarylaethylene oxide phosphate	-	-	3.0	-	-	-	-	-	-
Ethylene glycol	8.0	-	8.0	8.0	8.0	8.0	8.0	8.0	3.0
Urea	-	8.0	-	-	-	-	-	-	-

TABLE 1 (Continued)

Composition	Example								
	1	2	3	4	5	6	7	8	9
Silica	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	0.8
Siliconic antifoam	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	-
Formaldehyde 38% solution	0.125	0.25	0.25	0.25	0.25	0.25	0.25	0.25	-
Xanthan gum	0.05	0.1	0.1	0.1	0.1	0.1	0.1	0.05	-
Water	QS	QS	QS	QS	QS	QS	QS	QS	QS

55 **Claims**

1. An aqueous suspension concentrate composition consisting of 5.0 % to 50.0 % pendimethalin having at least 4 % and up to 100 % orange crystal form; 3.0 to 30.0 % coformulants and 20.0 % to 92.0 %

- water, obtainable by admixing, on a weight to volume basis, 5.0 % to 50.0 % pendimethalin having 4 % orange crystal form to 96 % yellow crystal form to 100 % orange crystal form; in an aqueous solution containing some of the coformulants such as a surfactant, a dispersing agent or wetting agent and an antifoaming agent; milling the resulting mixture, wherein the average particle size of suspended particles is brought to less than 20  $\mu\text{m}$ ; admixing the remaining coformulants, such as a thickening agent, a suspending agent, an antifreezing agent, a preservative and an additional surfactant; and packaging the resulting aqueous suspension concentrate composition.
2. A composition according to Claim 1, wherein said surfactants, dispersing agents or wetting agents are ethylene oxide/propylene oxide condensates; alkyl, aryl- and aryl, aryl-ethoxylates; lignosulfonates; cresol-formaldehyde condensates and sulfonates thereof, naphthalene-formaldehyde condensates and sulfonates thereof; polycarboxylates and derivatives; and mixtures thereof.
  3. A composition according to Claim 2, wherein said suspending agents are polysaccharide gums or cellulose derivatives.
  4. A composition according to Claim 3, wherein said polysaccharide gums are Xanthan gum, guar gum, gum arabic, and mixtures thereof.
  5. A composition according to Claim 4, wherein said antifreezing agents are ethylene glycol, propylene glycol, glycerine, urea, and mixtures thereof.
  6. A composition according to Claim 1, wherein said thickening agents are clays, precipitated silicas, polyvinyl alcohol, polyvinylpyrrolidone, polyacrylamides, and mixtures thereof.
  7. A composition according to Claim 1, wherein said preservative is a 38% formaldehyde solution, methyl or propyl parahydroxybenzoate, 2-bromo-2-nitro-propane-1,3-diol, sodium benzoate, glutaraldehyde, O-phenylphenol, benzisothiazolinones, 5-chloro-2-methyl-4-isothiazolin-3-one, pentachlorophenol, 2,4-dichlorobenzylalcohol, or mixtures thereof.
  8. A composition according to Claim 1, wherein said antifoaming agent is a silicic antifoaming agent.
  9. A composition according to Claim 1, comprising, on a weight to volume basis: 5.0% to 50.0% pendimethalin with 10%, on a weight to weight basis, of the orange crystal form; cresol-formaldehyde condensates and sulfonates thereof, naphthalene-formaldehyde condensates and sulfonates thereof, lignosulfonates, and mixtures thereof; 0.05% to 2.5% Xanthan gum, guar gum, gum arabic, or cellulose derivatives; 2.0% to 15.0% ethylene glycol, propylene glycol or urea; about 0.05% to 1.0% antifoaming agent; 0.05% to 2.0% thickening agent; 0.05% to 2.5% preservative; and the remainder water to total said composition to 100%.
  10. A composition according to Claim 1, comprising, on a weight to volume basis: 20.0% to 40.0% pendimethalin; 3.0% to 5.0% sodium cresol-formaldehyde condensate or sodium cresol-formaldehyde sulphonated condensate; 5% to 10% ethylene glycol or urea; 0.1% to 1.0% silicic antifoaming agent; 0.1% to 0.3% Xanthan gum; 0.2% to 1.0% of a 38% formaldehyde solution; and 48.3% to 79.4% water.
  11. A composition according to Claim 10, comprising, on a weight to volume basis: 40.0% pendimethalin; 5.0% sodium cresol-formaldehyde sulphonated condensate; 8.0% ethylene glycol; 0.5% silicic antifoaming agent; 0.2% Xanthan gum; 0.5% of a 38% formaldehyde solution; and 51.4% water.
  12. A method for the preparation of a stable aqueous suspension concentrate composition of pendimethalin, said method comprising: admixing, on a weight to volume basis, 5.0% to 50.0% pendimethalin having 4% orange crystal form to 96% yellow crystal form to 100% orange crystal form; an aqueous solution containing surfactant(s), dispersing agent(s) or wetting agent(s) and antifoaming agents; milling the resulting mixture, wherein the average particle size of suspended particles is less than 20  $\mu\text{m}$ ; admixing thickening agents, suspending agents, antifreezing agents, preservatives and additional surfactants; and packaging the resulting aqueous suspension concentrate composition.

13. A method according to Claim 12, wherein said orange crystal form is 10% orange crystal form to 90% yellow crystal form to 100% orange crystal form.

14. A method according to Claim 12, wherein said suspended particle size is 2  $\mu\text{m}$  to 10  $\mu\text{m}$ .

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# Patentansprüche

1. Eine Zusammensetzung eines wässrigen Suspensionskonzentrats, bestehend aus 5,0% bis 50,0% Pendimethalin mit mindestens 4% und bis zu 100% orangefarbener Kristallform, 3,0 bis 30,0% Koformulierungsmitteln und 20,0% bis 92,0% Wasser, erhältlich durch Vermischen, auf einer Gewichts- zu Volumen-Basis, von 5,0% bis 50,0% Pendimethalin mit 4% orangefarbener Kristallform und 96% gelber Kristallform bis 100% orangefarbener Kristallform, in einer wässrigen Lösung, die einige der Koformulierungsmittel enthält, wie ein oberflächenaktives Mittel, ein Dispersionsmittel oder ein Benetzungsmittel und ein Antischäummittel, Mahlen der resultierenden Mischung, worin die mittlere Teilchengröße der suspendierten Teilchen auf weniger als 20  $\mu\text{m}$  gebracht wird, Zumischen der übrigen Koformulierungsmittel, wie eines Verdickungsmittels, eines Suspensionsmittels, eines Gefrierschutzmittels, eines Konservierungsmittels und eines zusätzlichen oberflächenaktiven Mittels und Verpacken der resultierenden Zusammensetzung des wässrigen Suspensionskonzentrates.
2. Eine Zusammensetzung nach Anspruch 1, worin die oberflächenaktiven Mittel, Dispersionsmittel oder Benetzungsmittel Ethylenoxid/Propylenoxid-Kondensate, Alkyl-, Aryl- und Aryl-, Aryl-Ethoxylate, Lignosulfonate, Cresol-Formaldehyd-Kondensate und Sulfonate davon., Naphthalin-Formaldehyd-Kondensate und Sulfonate davon, Polycarboxylate und Derivate und Mischungen davon sind.
3. Eine Zusammensetzung nach Anspruch 2, worin die Suspensionsmittel Polysaccharide oder Cellulose-derivate sind.
4. Eine Zusammensetzung nach Anspruch 3, worin die genannten Polysaccharide Xanthan, Guar-Mehl, Gummi arabicum und deren Mischungen sind.
5. Eine Zusammensetzung nach Anspruch 4, worin die Gefrierschutzmittel Ethylenglycol, Propylenglycol, Glycerin, Harnstoff und deren Mischungen sind.
6. Eine Zusammensetzung nach Anspruch 1, worin die Verdickungsmittel Tone, gefällte Siliziumdioxide, Polyvinylalkohol, Polyvinylpyrrolidon, Polyacrylamide und deren Mischungen sind.
7. Eine Zusammensetzung nach Anspruch 1, worin das genannte Konservierungsmittel eine 38%-ige Formaldehydlösung, Methyl- oder Propyl-Parahydroxybenzoat, 2-Brom-2-nitropropan-1,3-diol, Natriumbenzoat, Glutaraldehyd, O-Phenylphenol, Benzisothiazolinone, 5-Chlor-2-methyl-4-isothiazolin-3-on, Pentachlorphenol, 2,4-Dichlorbenzylalkohol oder deren Mischungen sind.
8. Eine Zusammensetzung nach Anspruch 1, worin das genannte Antischäummittel ein Silikono-Antischäummittel ist.
9. Eine Zusammensetzung nach Anspruch 1, umfassend, auf einer Gewichts- zu Volumen-Basis: 5,0% bis 50,0% Pendimethalin mit 10%, auf einer Gewichts- zu Gewichts-Basis, der orangefarbenen Kristallform, Cresol-Formaldehyd-Kondensate und deren Sulfonate, Naphthalin-Formaldehyd-Kondensate und deren Sulfonate, Lignosulfonate und deren Mischungen, 0,05% bis 2,5% Xanthan, Guar-Mehl, Gummi arabicum oder Cellulose-derivate, 2,0% bis 15,0% Ethylenglycol, Propylenglycol oder Harnstoff, etwa 0,05% bis 1,0% Antischäummittel, 0,05% bis 2,0% Verdickungsmittel, 0,05% bis 2,5% Konservierungsmittel und der Rest ist Wasser bis zu 100% der genannten Zusammensetzung.
10. Eine Zusammensetzung nach Anspruch 1, umfassend, auf einer Gewichts- zu Volumen-Basis: 20,0% bis 40,0% Pendimethalin, 3,0% bis 5,0% Natriumcresol-Formaldehyd-Kondensat oder sulfoniertes Natriumcresol-Formaldehyd-Kondensat, 5% bis 10% Ethylenglycol oder Harnstoff, 0,1% bis 1,0% Silikono-Antischäummittel, 0,1% bis 0,3% Xanthan, 0,2% bis 1,0% einer 38%-igen Formaldehydlösung und 48,3% bis 79,4% Wasser.

11. Eine Zusammensetzung nach Anspruch 10, umfassend, auf einer Gewichts- zu Volumen-Basis: 40,0% Pendimethalin, 5,0% sulfoniertes Natriumcresol-Formaldehyd-Kondensat, 8,0% Ethylenglycol, 0,5% Silikono-Antischäummittel, 0,2% Xanthan, 0,5% einer 38%-igen Formaldehydlösung und 51,4% Wasser.

12. Ein Verfahren zur Zubereitung einer stabilen Zusammensetzung eines wässrigen Suspensionskonzentrats von Pendimethalin, wobei das genannte Verfahren umfaßt: Vermischen, auf einer Gewichts- zu Volumen-Basis, von 5,0% bis 50,0% Pendimethalin mit 4% orangefarbener Kristallform zu 96% gelber Kristallform bis 100% orangefarbener Kristallform, eine wässrige Lösung, die oberflächenaktive(s) Mittel, Dispersionsmittel oder Benetzungsmittel und Antischäummittel enthält, Mahlen der resultierenden Mischung, worin die mittlere Teilchengröße der suspendierten Teilchen geringer als 20 µm ist, Hinzumischen von Verdickungsmitteln, Suspensionsmitteln, Gefrierschutzmitteln, Konservierungsmitteln und zusätzlichen oberflächenaktiven Mitteln und Verpacken der resultierenden Zusammensetzung des wässrigen Suspensionskonzentrats.

13. Ein Verfahren nach Anspruch 12, worin die orangefarbene Kristallform 10% orangefarbene Kristallform zu 90% gelbe Kristallform bis 100% orangefarbene Kristallform ist.

14. Ein Verfahren nach Anspruch 12, worin die suspendierte Teilchengröße 2 µm bis 10 µm beträgt.

## Revendications

1. Composition aqueuse concentrée sous forme de suspension, constituée de 5,0 % à 50,0 % de pendiméthaline comprenant au moins 4 % et jusqu'à 100 % de forme cristalline orangée ; 3,0 à 30,0 % d'adjuvants de formulation et 20,0 % à 92,0 % d'eau, qui peut être obtenue en mélangeant, comme exprimé en poids par volume, 5,0 % à 50,0 % de pendiméthaline comprenant de 4 % de forme cristalline orangée pour 96 % de forme cristalline jaune à 100 % de forme cristalline orangée, dans une solution aqueuse contenant certains des adjuvants de formulation tels qu'un agent tensio-actif, un agent dispersant ou un agent mouillant et un agent antimousse ; en broyant le mélange résultant, la grosseur moyenne des particules en suspension étant amenée à moins de 20 µm ; en ajoutant et mélangeant les adjuvants de formulation restants, tels qu'un épaississant, un agent suspendant, un antigel, un conservateur et un agent tensio-actif supplémentaire ; et en conditionnant sous emballage la composition aqueuse concentrée sous forme de suspension résultante.

2. Composition selon la revendication 1, dans laquelle lesdits agents tensio-actifs, agents dispersants ou agents mouillants sont des produits de condensation oxyde d'éthylène/oxyde de propylène ; des éthoxylats d'alkyle, aryle et arylaryle et leurs dérivés ; des lignosulfonates ; des produits de condensation crésol-formaldéhyde et leurs sulfonates ; des produits de condensation naphthalène-formaldéhyde et leurs sulfonates ; des polycarboxylats et leurs dérivés ; et leurs mélanges.

3. Composition selon la revendication 2, dans laquelle lesdits agents suspendants sont des gommes de polysaccharide ou des dérivés de cellulose.

4. Composition selon la revendication 3, dans laquelle lesdites gommes de polysaccharide sont la gomme de xanthane, la gomme guar, la gomme arabique et leurs mélanges.

5. Composition selon la revendication 4, dans laquelle lesdits antigels sont l'éthylène-glycol, le propylène-glycol, la glycérine, l'urée et leurs mélanges.

6. Composition selon la revendication 1, dans laquelle lesdits épaississants sont des argiles, des silices précipitées, l'alcool polyvinylique, la polyvinylpyrrolidone, des polyacrylamides et leurs mélanges.

7. Composition selon la revendication 1, dans laquelle ledit conservateur est une solution de formaldéhyde à 38 %, le para-hydroxybenzoate de méthyle ou de propyle, le 2-bromo-2-nitropropane-1,3-diol, le benzoate de sodium, le glutaraldéhyde, le O-phénylphénol, des benzisothiazolinones, la 5-chloro-2-méthyl-4-isothiazoline-3-one, le pentachlorophénol, l'alcool 2,4-dichlorobenzyle ou leurs mélanges.

8. Composition selon la revendication 1, dans laquelle ledit agent antimousse est un agent antimousse du type silicone.

5 9. Composition selon la revendication 1, comprenant, comme exprimé en poids par volume : 5,0 % à 50,0 % de pendiméthaline dont une fraction de 10 % en poids est de la forme cristalline orangée ; des produits de condensation crésol-formaldéhyde et leurs sulfonates, des produits de condensation naphthalène-formaldéhyde et leurs sulfonates, des lignosulfonates, et leurs mélanges ; 0,05 % à 2,5 %  
10 de gomme de xanthane, gomme guar, gomme arabique ou dérivés de cellulose ; 2,0 % à 15,0 % d'éthylène-glycol, propylène-glycol ou urée ; environ 0,05 % à 1,0 % d'agent antimousse ; 0,05 % à 2,0 % d'épaississant ; 0,05 % à 2,5 % de conservateur ; et le reste d'eau pour compléter ladite composition à 100 %.

15 10. Composition selon la revendication 1, comprenant, comme exprimé en poids par volume : 20,0 % à 40,0 % de pendiméthaline ; 3,0 % à 5,0 % de produit de condensation crésol-formaldéhyde sodique ou de produit de condensation crésol-formaldéhyde sulfoné sodique ; 5 % à 10 % d'éthylène-glycol ou d'urée ; 0,1 % à 1,0 % d'agent antimousse du type silicone ; 0,1 % à 0,3 % de gomme de xanthane ; 0,2 % à 1,0 % d'une solution de formaldéhyde à 38 % ; et 48,3 % à 79,4 % d'eau.

20 11. Composition selon la revendication 10, comprenant, comme exprimé en poids par volume : 40,0 % de pendiméthaline ; 5,0 % de produit de condensation crésol-formaldéhyde sulfoné sodique ; 8,0 % d'éthylène-glycol ; 0,5 % d'agent antimousse du type silicone ; 0,2 % de gomme de xanthane ; 0,5 d'une solution de formaldéhyde à 38 % ; et 51,4 % d'eau.

25 12. Procédé pour la préparation d'une composition aqueuse concentrée stable de pendiméthaline sous forme de suspension, ledit procédé consistant à : mélanger, comme exprimé en poids par volume, 5,0 % à 50,0 % de pendiméthaline comprenant d'au moins 4 % de forme cristalline orangée pour 96 % de forme cristalline jaune à 100 % de forme cristalline orangée, dans une solution aqueuse contenant un ou plusieurs agents tensio-actifs, agents dispersants ou agents mouillants et des agents antimousse ; broyer le mélange résultant, la grosseur moyenne des particules en suspension étant inférieure à 20  
30  $\mu\text{m}$  ; ajouter et mélanger des épaississants, agents suspendants, antigels, conservateurs et agents tensio-actifs supplémentaires ; et conditionner sous emballage la composition aqueuse concentrée sous forme de suspension résultante.

35 13. Procédé selon la revendication 12, dans lequel ladite forme cristalline orangée représente de 10 % de forme cristalline orangée pour 90 % de forme cristalline jaune à 100 % de forme cristalline orangée.

14. Procédé selon la revendication 12, dans lequel ladite grosseur des particules en suspension est de 2  $\mu\text{m}$  à 10  $\mu\text{m}$ .

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